REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY *(Leave blank)*	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED	
	31 Jul 97	Final	1 May 96 - 30 Apr 98

4. TITLE AND SUBTITLE
"Fracture Toughness and Processing Routes Relations in Commercial Titanium Alloys for Developing Alternative Alloys for Ti-6AI-4V"

5. FUNDING NUMBERS
G - F49620-96-1-0183

6. AUTHOR(S)

Professor Mitsuo Niinomi

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Japan Titanium Society
Daishin Bldg., 4F, 9, 2-Chome, Kanda
Nishiki-Cho, Chiyoda-Ku
Tokyo Japan 101

8. PERFORMING ORGANIZATION REPORT NUMBER
N/A

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Asian Office of Aerospace Research and Development (AOARD)
Unit 45002
APO AP 96337-5002

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
AOARD 96-04

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION AVAILABILITY STATEMENT
Approved for public release; distribution is unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

The static and dynamic fracture toughness, strength, and ductility of various titanium alloys were investigated. The crack propagation toughness and fatigue crack propagation rate under both static and dynamic conditions were also investigated and compared with that of T-6AI-4V at the elevated temperatures. The results showed that sudden decrease in strength, fracture toughness values or the stress intensity factors were noticeable at the elevated temperature of 1123K. The fracture toughness values evaluated from the load values (stress intensity factors) also decreased at a single annealing temperature of 1123K. The sudden decrease in strength was recovered by conducting duplex annealing. By duplex annealing, Widmanstatten alpha precipates in beta and then beta was able to stabilize the strength.

14. SUBJECT TERMS
Titanium, fracture toughness, ductility, crack propagation, duplex annealing, Widmanstatten

15. NUMBER OF PAGES
19

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	UL

Final report

on "Fracture Toughness and Processing Routes relations in Commercial Titanium Alloys for Developing Alternative Alloys for Ti-6Al-4V".

Grant No.F4960-96-1-0183

Mitsuo Niinomi

Professor

Toyohasi University of Technology

31 July 1997

(Follow instructions on the back)

1. Federal Agency and Organizational Element to Which Report is Submitted	2. Federal Grant or Other Identifying Number Assigned By Federal Agency	OMB Approval No.	Page	of
UNITED STATES AIR FORCE	F49620-96-1-0183	0348-0039	1	1 pages

3. Recipient Organization (Name and complete address, including ZIP code)

Japan Titanium Society

Daishin Bldg. 4F, 9, 2-Chome, Kanda, Nishiki-Cho, Chiyoda-Ku, Tokyo, Japan 101

4. Employer Identification Number	5. Recipient Account Number or Identifying Number	6. Final Report	7. Basis
	CFDA #12.800	☑ Yes ☐ No	☑ Cash ☐ Accrual

8. Funding/Grant Period (See instructions) From: (Month, Day, Year)	To: (Month, Day, Year)	9. Period Covered by this Report From: (Month, Day, Year)	To: (Month, Day, Year)
May 1 96	Apr 30 97	May 1 96	Apr 30 97

10. Transactions:	I Previously Reported	II This Period	III Cumulative
a. Total outlays			$ 25,000
b. Recipient share of outlays			$ 0
c. Federal share of outlays			$ 0
d. Total unliquidated obligations			$ 0
e. Recipient share of unliquidated obligations			$ 0
f. Federal share of unliquidated obligations			$ 0
g. Total Federal share (Sum of lines c and f)			$ 0
h. Total Federal funds authorized for this funding period			$25,000
i. Unobligated balance of Federal funds (Line h minus line g)			$ 0

11. Indirect Expense

a. Type of Rate (Place "X" in appropriate box) ☐ Provisional ☐ Predetermined ☐ Final ☐ Fixed

b. Rate	c. Base	d. Total Amount	e. Federal Share

12. *Remarks: Attach any explanations deemed necessary or information required by Federal sponsoring agency in compliance with governing legislation.*

13. Certification: **I certify to the best of my knowledge and belief that this report is correct and complete and that all outlays and unliquidated obligations are for the purposes set forth in the award documents.**

Typed or Printed Name and Title	Telephone (Area code, number and extension)
Mitsuo Niinomi, Professor	+81-532-44-6706
Signature of Authorized Certifying Official: 新家光雄 Niinomi	Date Report Submitted: 31 July 1997

STATEMENT OF WORK

Selected U.S. and Japanese made titanium alloys of Ti - 62222 (U.S. alloy) and SP - 700 (Japanese alloy), respectively are alternative lower cost alloys to Ti - 6Al - 4V will be processed under various conditions to change the microstructure. The processes for changing the microstructures will be forging, rolling and heat treatment.

The static fracture toughness, J_{IC} or K_{IC}, dynamic fracture toughness, J_{Id}, strength and ductility of the variously processed alloys will be investigated. The fracture toughness and crack propagation toughness, T_{mat}, under both static and dynamic conditions and strength balances of the alloys will be also investigated to select the alloys and alloy conditions which show the greater balance of fracture toughness and strength comparing with that of Ti - 6Al -4V.

The fatigue crack propagation rate will be also investigated from the same point of view as stated above.

The fatigue precracked three point bending specimens, size of 10 x 10 x 55 mm,will be used for both static and dynamic fracture toughness tests. The static fracture toughness tests will be carried out using an Instron type testing machine at a cross head speed of 0.3 mm/ min in air. The dynamic fracture toughness tests will be carried out using a computer aided instrumented Charpy impact testing system, CAI system, at an impact speed of over 1.0 m/ sec in air. The crack initiation point will be detected using compliance changing rate method, electrical potential method or acoustic emission (AE) method, in static fracture toughness tests and will be detected using compliance changing rate method in dynamic fracture toughness tests. The fracture toughness values will be calculated by Rice's equation. The crack propagation toughness is also determined using key curve method or electrical potential method.

CT specimens , size of 50 x 60 x 10mm, will be used for evaluating fatigue crack propagation rate. Fatigue crack propagation tests will be performed on the CT specimens using electro - servo - hydraulic machine with a load ratio, R of 0.1 and a frequency of 20 Hz (sine wave). A constant - load - amplitude method Of ASTM E - 647 88 a will be adopted in the higher fatigue crack growth region above 10^{-8} m/cycle. Cyclic stress intensity factor range, ΔK, decreasing method will be adopted in the lower fatigue crack growth region than 10^{-8} m/cycle. Fatigue crack length measurements will be done on the specimen surface using a light microscope. Crack closure measurements will be carried out using back - face strain.

The round bar specimens, size of ϕ 4 x 55 mm will be used for tensile tests. The tensile tests for

evaluating the strength and ductility, that is, 0.2 % proof stress, ultimate tensile strength, elongation and reduction of area will be carried out using an Instron type testing machine at a cross head speed of 0.5 mm/min in air.

The fracture surfaces will be characterized using a scanning electron microscope, SEM, for analyzing the fracture mechanisms of the alloys. The fracture surface parameters like fracture surface roughness, the number of the microcracks, the number of secondary cracks effective crack length, size of dimples and so on. The fracture surface parameters will be related to the fracture toughness values and fatigue crack propagation rate. The cross section of the fracture surface will be characterized using a light microscope and a SEM to clarify the crack propagation path.

The analysis of three dimensional fracture mechanisms of the alloys will be done using an AE analysis in static fracture toughness tests.

The microstructures of the alloys will be characterized using a light microscope, SEM, transmission electron microscope, TEM. The microstructural parameters like prior β grain size, inter spacing of α lath, colony size, volume fraction of precipitated α, nearest neighbors distance, volume faction of precipitates and so on will be measured. Further more the dislocation structure near the fracture surface will be also characterized using a TEM.

The fracture toughness and crack propagation toughness under both static and dynamic conditions, fatigue crack propagation rate, strength, ductility and microstructure relations will be quantitatively analyzed. The process conditions which will give the best microstructure for the best balance of fracture toughness, fatigue crack propagation resistance and strength will be identified.

The proper process conditions will be also discussed from the viewpoint of process cost. Finally the possible alternative alloy for Ti - 6Al - 4V alloy and the possibility of the low cost processing conditions for the alloy will be clearly understood.

This program will not only use Japanese and U. S. developed new alloys, but it will also incorporate the work of Japanese and U. S. university research personnel, so that the study will induce research and evaluation criteria which of use in the two countries.

This program will involve two Japanese Co - PI, one U. S. Co - PI and two Japanese graduate students. In addition to developing very useful processing data, this program also will serve as a vehicle to promote closer international materials cooperation in such an important are

Status of Effort

The plates with 50 mm and 12 mm thick of SP-700 and the plates with about 64mm and 13 mm of Ti-62222S have been received after long delay. The experimental schedule is very much delayed because of long time delay of materials fabrication.. The study on the SP-700 has been started firstly because SP-700 has received fairly earlier than Ti-62222S.

The study on the fracture toughness on SP-700 has been started firstly. Some data on the tensile properties, hardness and fracture toughness have been gotten. Those will be reported in the report documents attached.

The study on the Ti-62222S has been just started. The heat treatment process is being checked right now.

Technical report on the fracture toughness investigation on SP-700

1. Purpose

According to the paper presented by NKK at 8th World Conference on Titanium, Oct.95, Birmingham, U.K.[1], The fracture toughness, K_{Ic}, shows sudden decrease when SP-700 is conducted with the single annealing where the annealing temperature is round 1123 K. The sudden decrease of K_{Ic} in SP-700 is recovered when the duplex annealing where first and second annealing temperatures are round 1123 K and 993 K, respectively is conducted as shown in Fig.0.

The cause for the sudden decrease in K_{Ic} by the single annealing has not been cleared. The fracture toughness tests , tensile tests , hardness tests and microstructural observations on SP-700 conducted with various annealing processes are therefore carried out to clarify the cause of sudden decrease of K_{Ic} associated with the single annealing in this study.

2. Experimental procedures

The samples with the size of 55 x 11x 11 mm were machined from the rolling plate with 13 mm thick so for the longitudinal direction of the samples as to be equal to the rolling direction of the plate. The samples were then conducted with the single annealing processes where the annealing temperatures were 1103 K (830°C), 1113 K (840°C), 1123 K (850°C), 1133 K (860°C) and 1143 K (870°C). The duplex annealing processes were also conducted on the other samples followed by the single annealing processes described above. The second annealing temperature was constant to be 993 K (720°C).The heat treated samples were machined to be tensile specimens with a gage diameter of 6 mm and a length of 55 mm, and static fracture toughness test specimens with a thickness of 10 mm, a width of 10 mm and a length of 55 mm. The fatigue pre-crack was introduced into the fracture toughness test specimen according to the ASTM E 813 [2].

Tensile tests were carried out using an Instron type testing machine at a cross head speed of 0.5 mm/min. The load was detected by a load cell in the testing machine and the strain was detected by both clip gage and foil gage directly attached to the specimen.

Three point bend Fracture toughness tests were carried out using an Instron type testing machine according to the ASTM E 813[2]. The J integral at the crack initiation point, J_{in}, was then evaluated. The crack initiation point was detected using an alternative current potential method.

Plane stain fracture toughness was also tried to be measured according to the ASTM E 399 [3]. K_{Ic} values were however not obtained in this study.

Vickers hardness was measured on small samples machined from the fracture toughness tested

specimens using a Vickers hardness tester.

Microstructural observations were done on the small samples machined from the fracture toughness tested specimens using a light microscope.

3. Results

3.1 Vickers hardness

Vickers hardness of the specimens conducted with the single and duplex annealing processes was shown in Fig.1 as a function of annealing temperature. The sudden decrease in Vickers hardness is obviously seen at an annealing temperature of 1123 K. The sudden decrease in Vickers hardness is recovered by duplex annealing.

3.2 Tensile properties

Ultimate tensile strength, 0.2 % proof stress, elongation and reduction of area were shown in Fig.s 2 (a), 2 (b), 3 (a) and 3 (b), respectively.

The ultimate strength and 0.2 % proof stress shows a sudden decrease at an annealing temperature of 1123 K. The sudden decrease in ultimate tensile strength and 0.2 % proof stress is recovered by the duplex annealing.

The elongation and reduction of ares at an annealing temperature of 1123 K are a little greater than those at other annealing temperatures. The elongation and reduction area shows nearly the same trend each other against annealing temperature.

3.3 Fracture toughness

Up to now only the fracture toughness values of the specimens conducted with single annealing processes are available.

Fracture toughness, K_Q, K_{in}, J_{Ic} and K_{JIc} are shown in Figs. 4 (a), 4 (b), 5 (a) and 5 (b) as a function of single annealing temperature.

K_Q and K_{in} are the non-valid stress intensity value obtained according to the ASTM E 399 [3[and stress intensity factor at the crack initiation point, respectively. J_{Ic} is the valid J integral at the crack initiation point according to the ASTM E 813 [2]. K_{JIc} is the converted K_{IC} from J_{Ic}.

J_{Ic} decreases against the single annealing temperature up to an single annealing temperature of 1113 K and then increases against the single annealing temperature. Other toughness values decrease up to a single annealing temperature of 1123 K and then increase against the single annealing temperature. The fracture toughness values show an minimum value around 1123 K.

3.4 Microstructures

Light micrographs of the specimens conducted with the single annealing processes are shown in Figs. 7, 8 and 9.

The fraction of equiaxed α is decrease with increasing annealing temperature. No remarkable change can be found on the light micrographs. SEM and TEM characterizations are furthermore needed.

4. Discussion

The sudden decrease in strength values has been found in this study at a single annealing temperature of 1123 K. The fracture toughness values evaluated from the load values, that is, stress intensity factors shows also sudden decrease at a single annealing temperature of 1123 K as well as shown in the K_{Ic} values against the single annealing temperature by NKK. J_{Ic} values also shows very low value at this temperature. The sudden decrease of fracture toughness in the specimens conducted with single annealing process is associated with the sudden decrease in the strength. However, the sudden decrease in strength is recovered by conducting duplex annealing. By duplex annealing, Widmanstatten α precipitates in β and then β is stabilized. The stability of β phase will therefore affect the strength of the specimens. Further investigations on the stability of β and microstructural observations on un-deformed and deformed specimens by SEM and TEM are needed.

The fracture toughness tests on the specimens conducted with duplex annealing are planning in the future study. The tensile tests and fracture toughness tests on the specimens conducted the following heat treatments are also planning in the future study ;

(1) 1123 K(850°C) x 1h/FC faster cooling rate
(2) 1123 K(850°C) x 1h/FC medium cooling rate
(3) 1123 K(850°C) x 1h/FC slowest cooling rate
(4) 1103 K(830°C) x 1h/FC medium cooling rate
(5) 1103 K(830°C)/ x 1h/FC slowest cooling rate

References

[1] A. Ogawa, K. Minakawa and S. Takagi : Proc. 8th World Conference on Titanium, (1995), pp.1251-1258.

[2] Standard Test Method for JIc, A measure of Fracture Toughness, ASTM Designation E 813-89, ASTM, Philadelphia, PA, 1989.

[3] Standard Test Method for Plane - Strain Fracture Toughness of Metallic Materials, ASTM Designation E 399 - 90, ASTM, Philadelphia, PA,

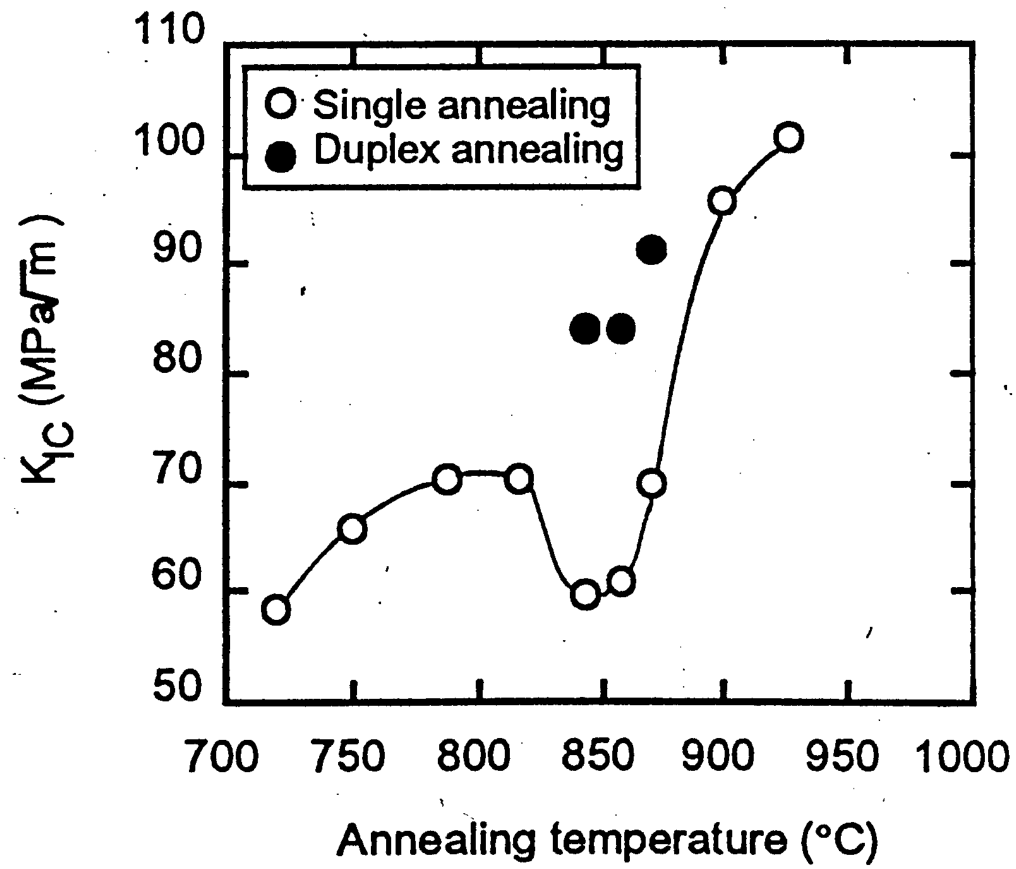


Fig. 0 Effect of annealing temperature K_{IC}.

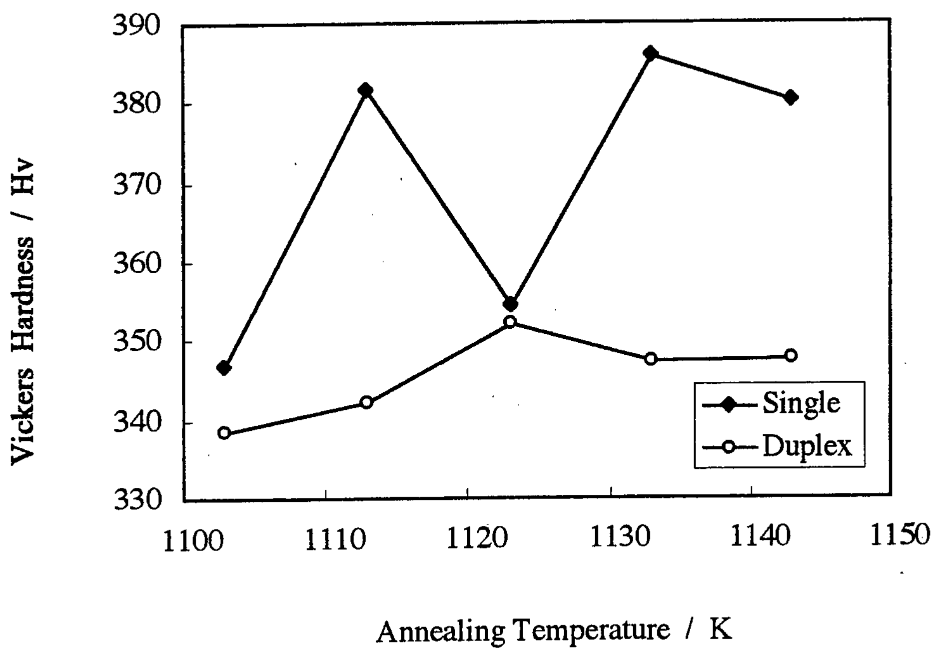
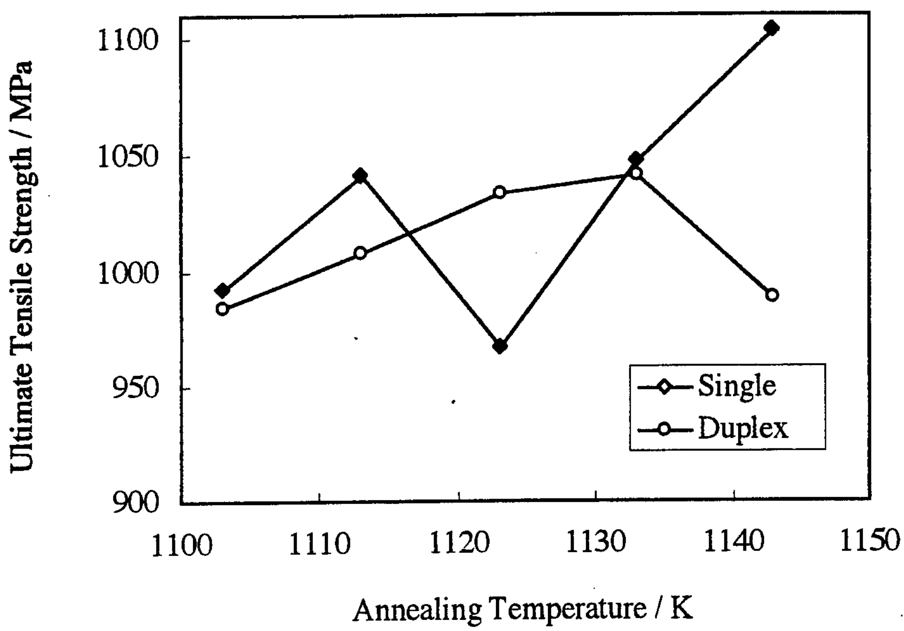
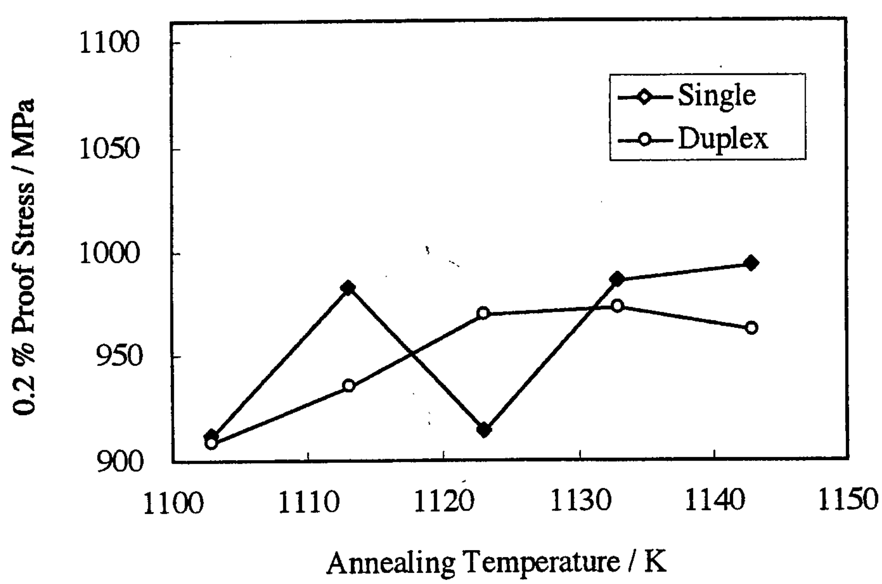


Fig.1 Effect of annealing temperature on Vickers hardness of SP-700.



(a)



(b)

Fig.2 Effect of annealing temperature on (a) ultimate tensile strength and (b) proof stress of SP-700

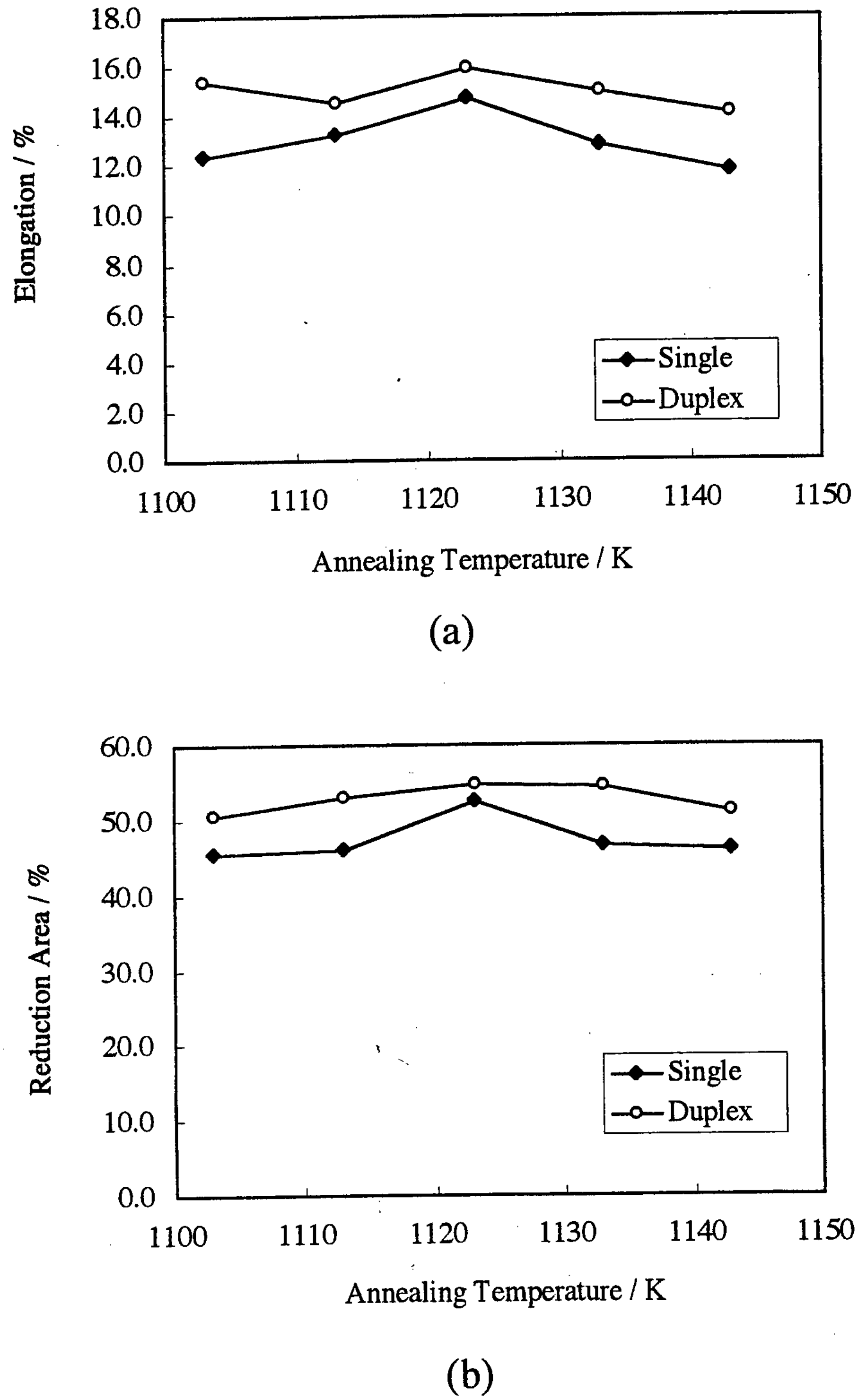


Fig. 3 Effect of annealing temperature on (a) elongation and (b) reduction area of SP-700.

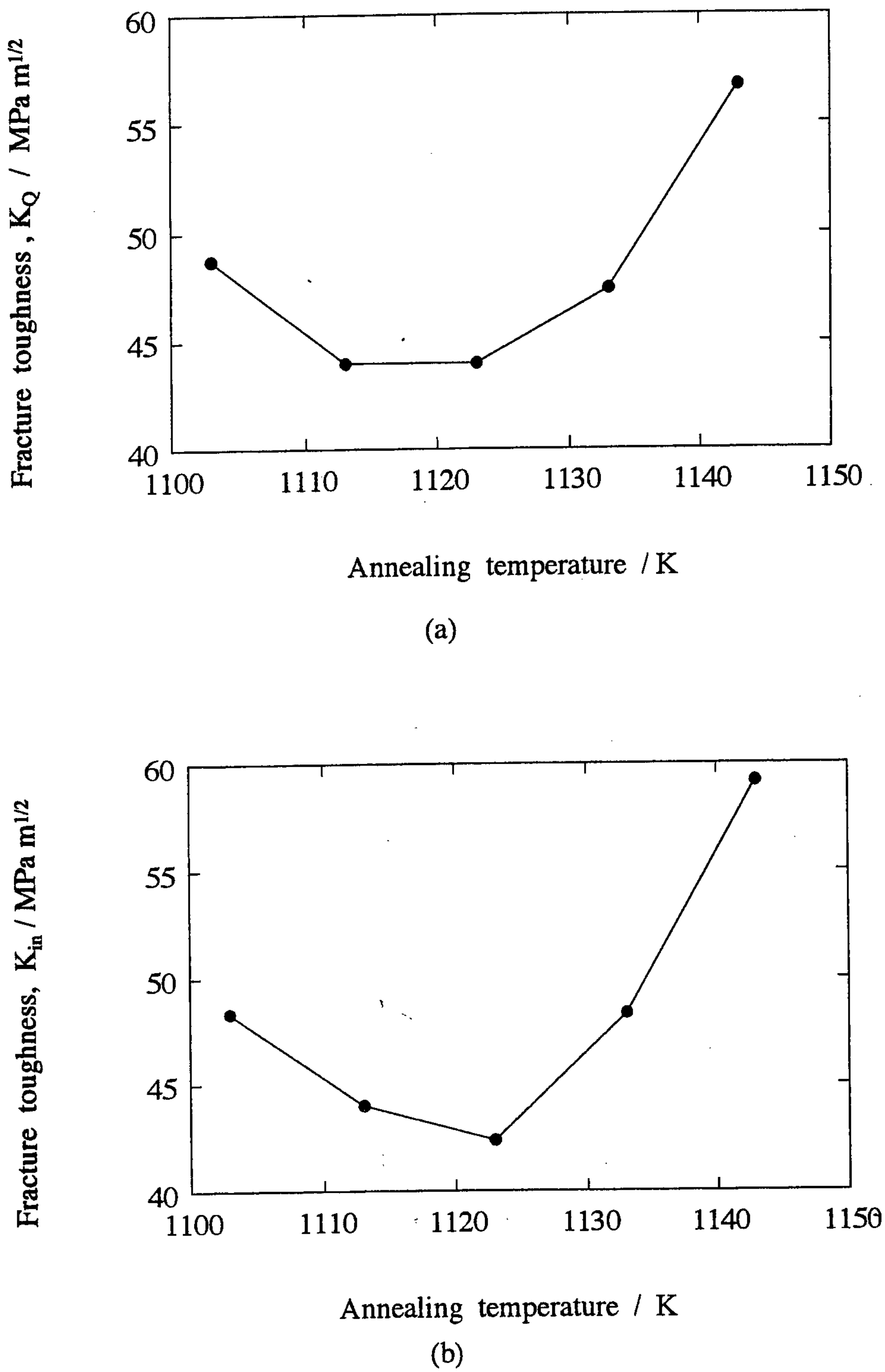


Fig.4 Effect of single-step annealing temperature on (a) K_Q and (b) K_{in} of SP-700.

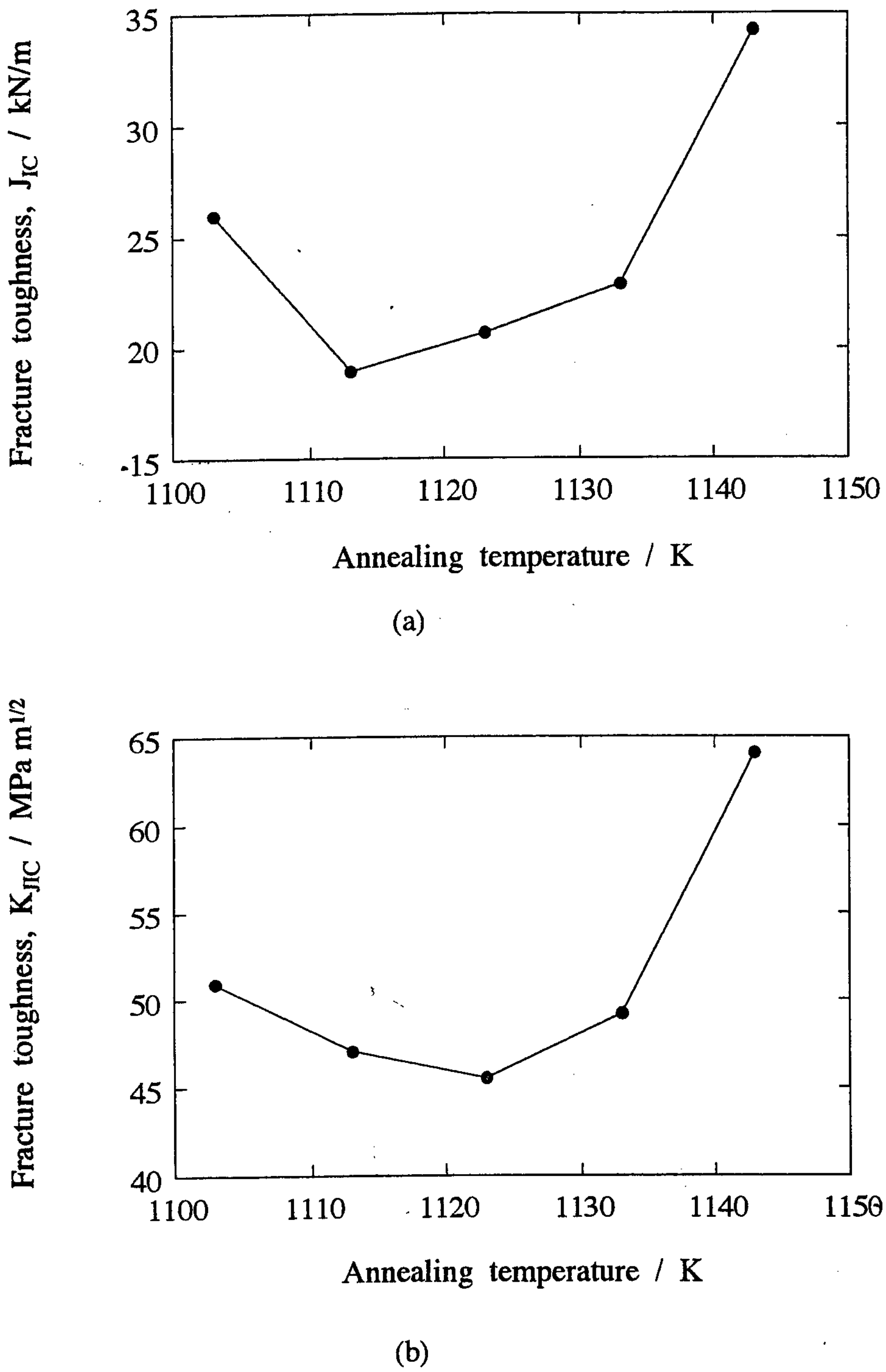


Fig.5 Effect of single annealing temperature on (a) J_{IC} and (b) K_{Ic} of SP-700.

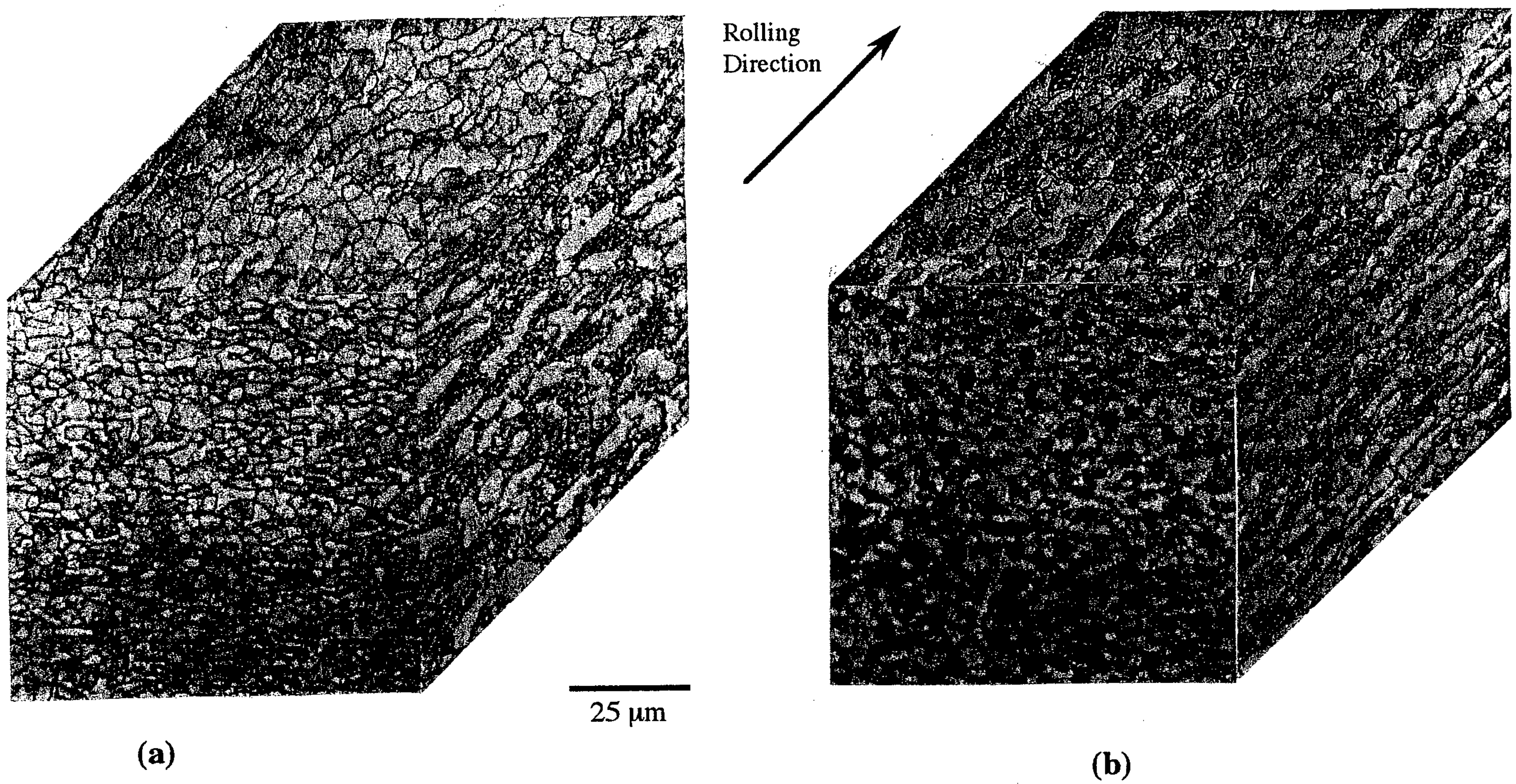


Fig. 6. Micrographs of (a) single annealed ; 1103K and
(b) duplex annealed ; 1103K + 993K in SP-700.

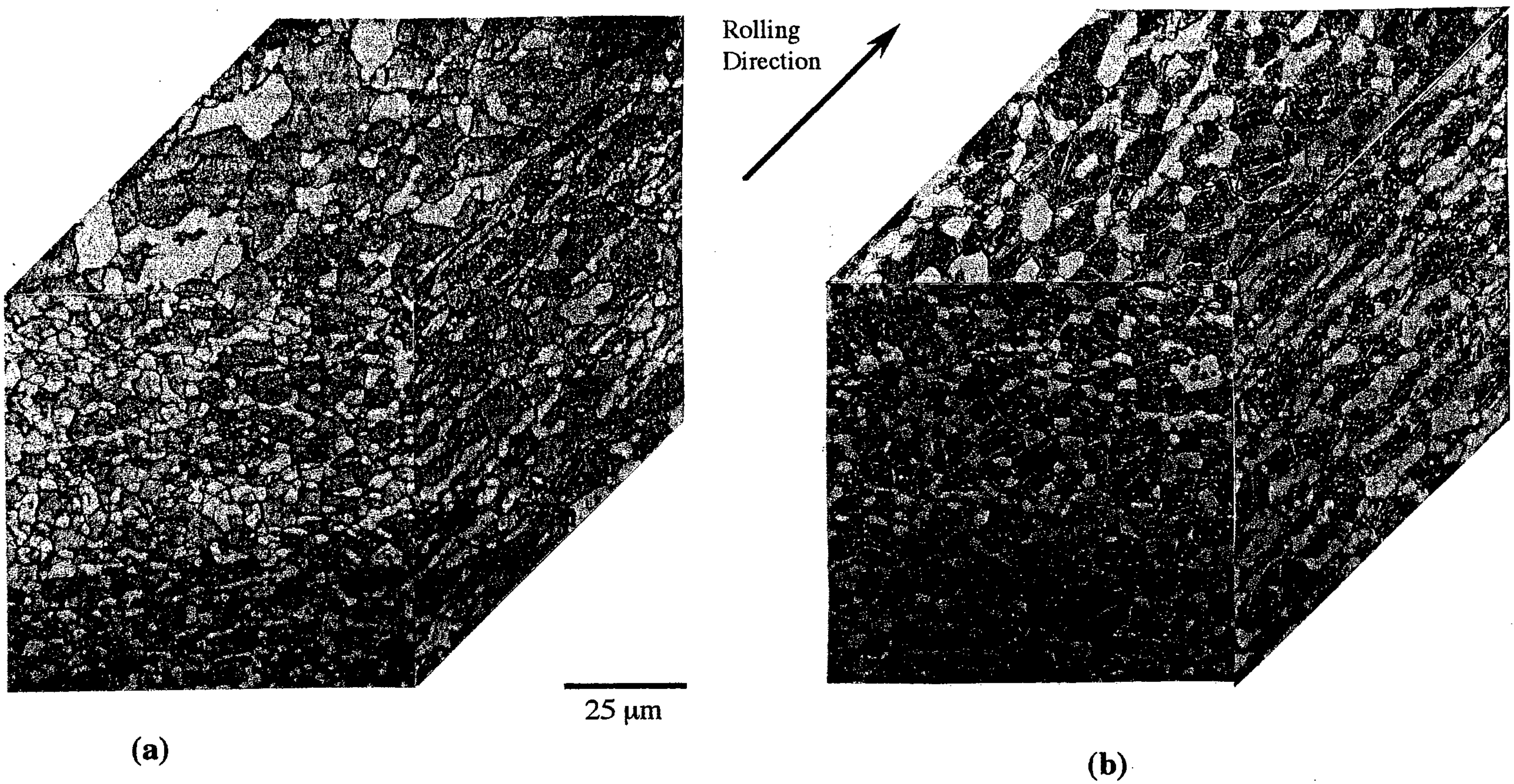


Fig. 7. Micrographs of (a) single annealed ; 1113K and

(b) duplex annealed ; 1113K + 993K in SP-700.

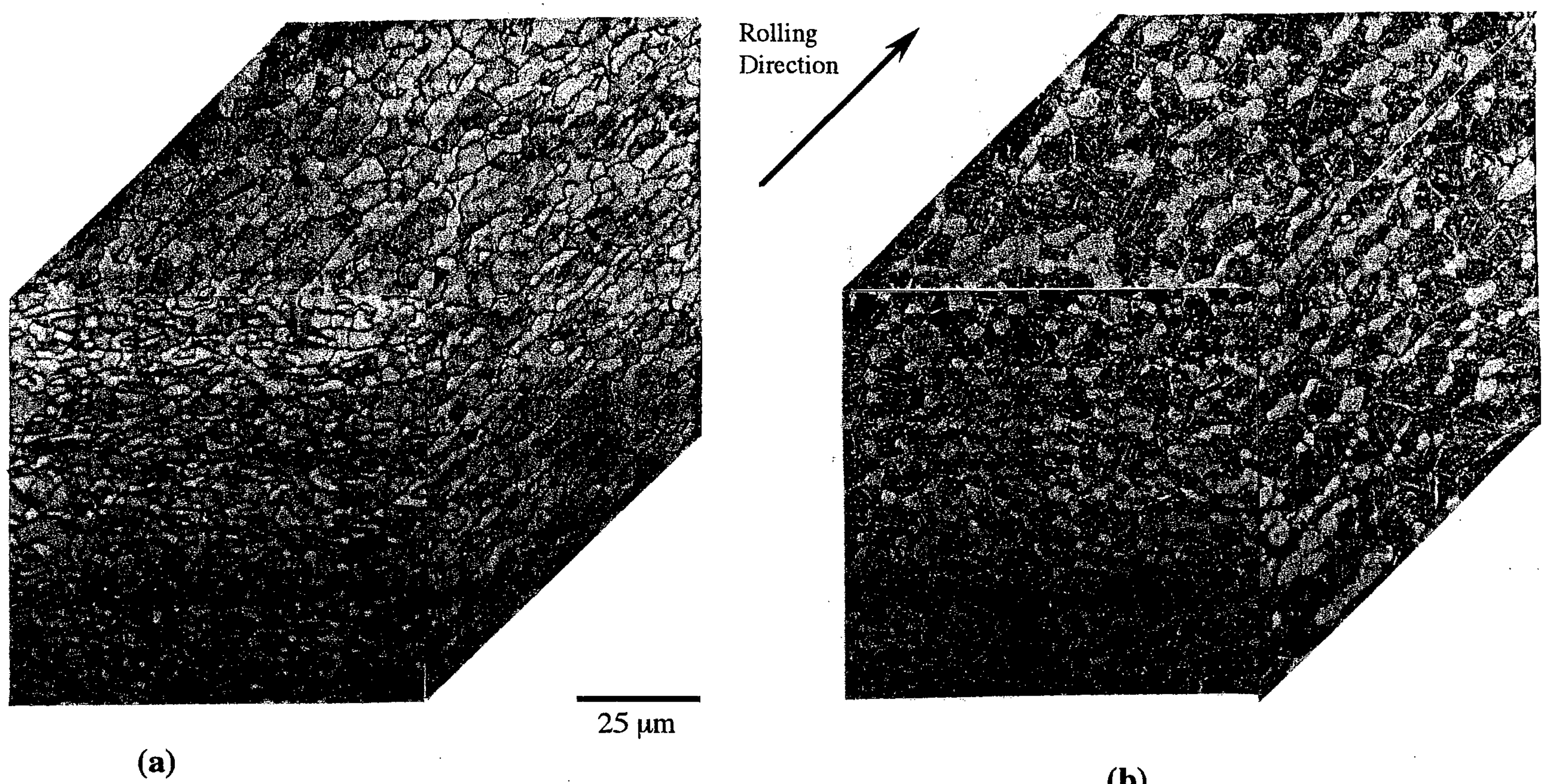


Fig. 8. Micrographs of (a) single annealed ; 1123K and (b) duplex annealed ; 1123K + 993K in SP-700.

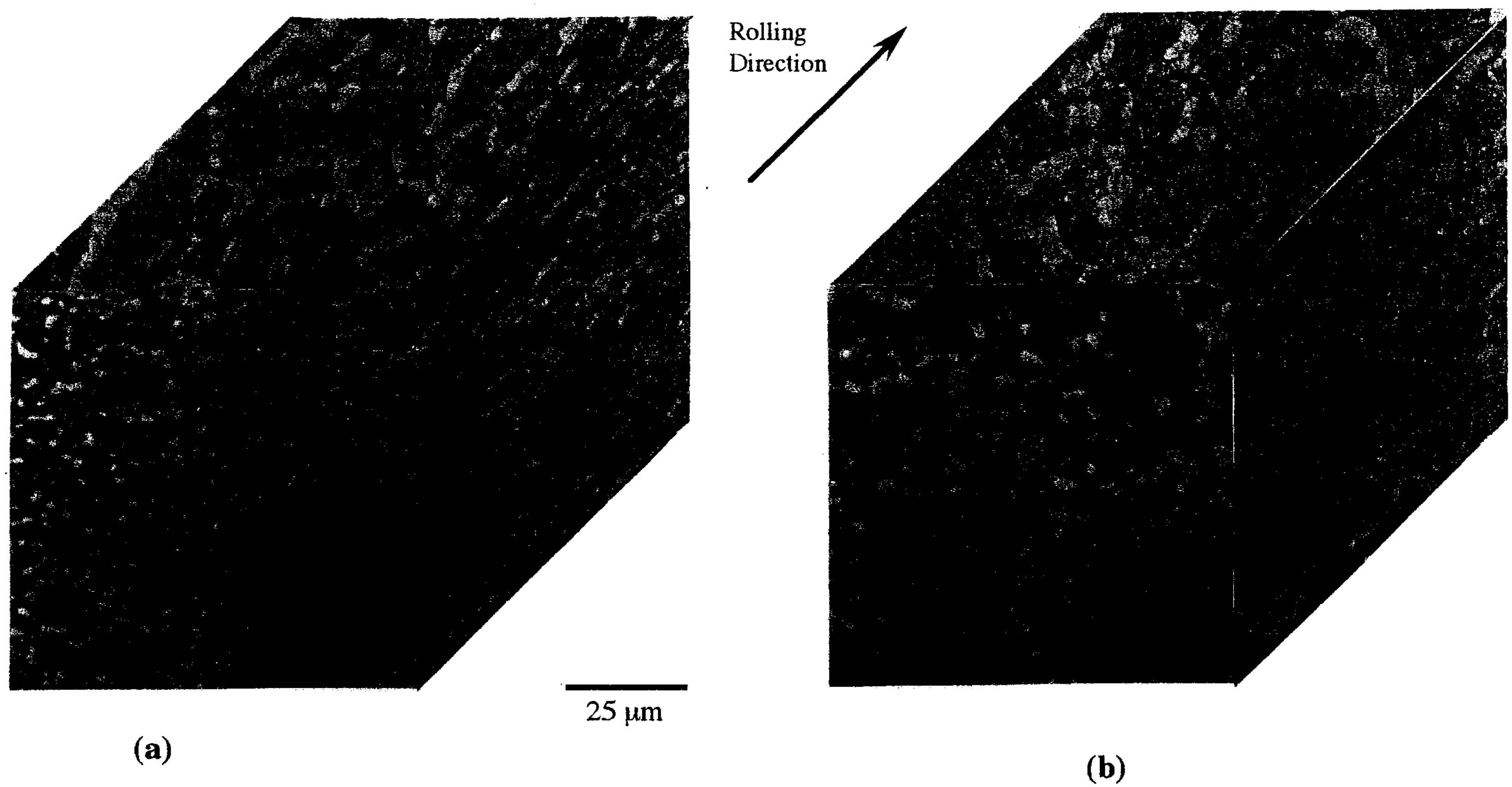


Fig. 9. Micrographs of (a) single annealed ; 1133K and (b) duplex annealed ; 1133K + 993K in SP-700.

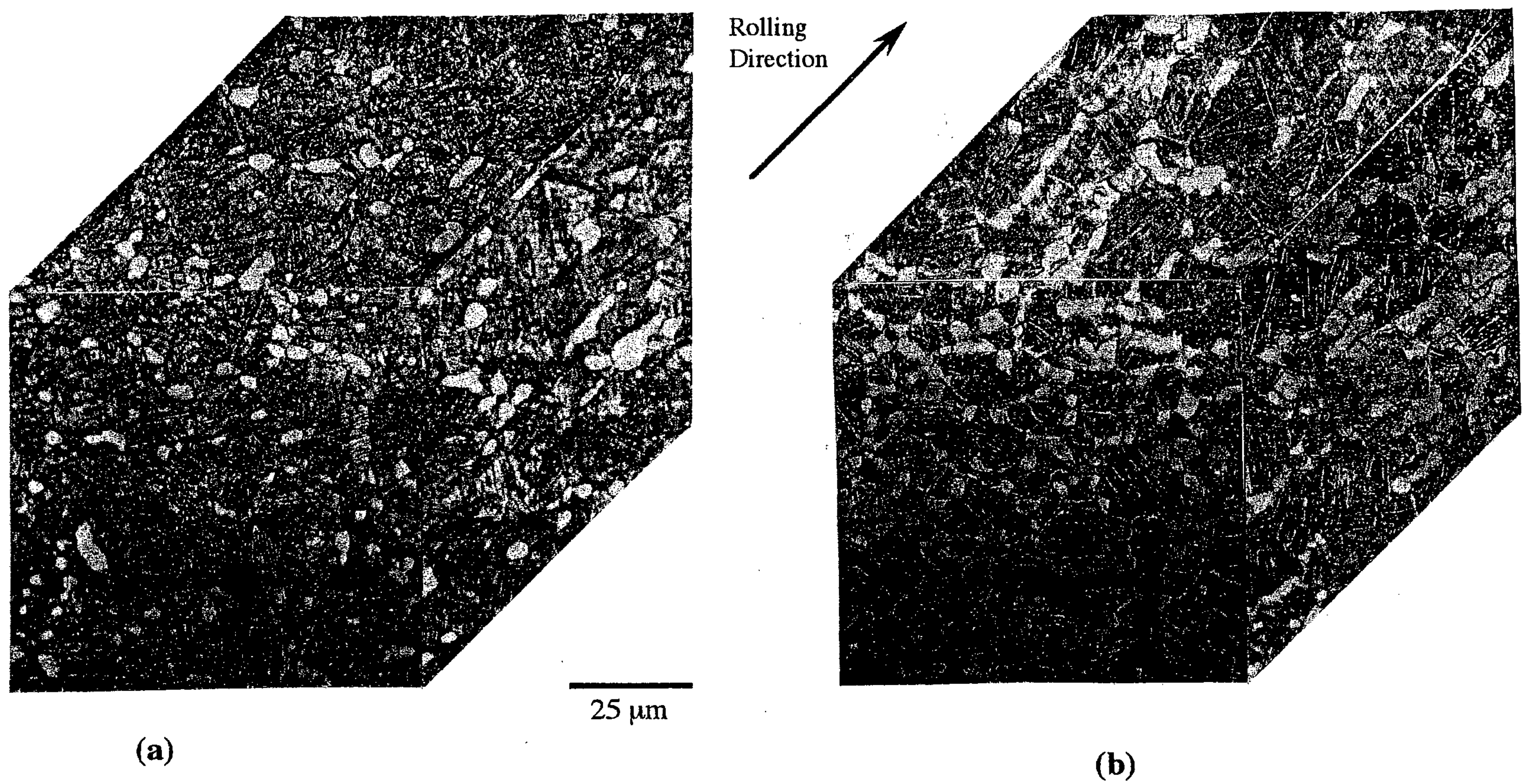


Fig. 10. Micrographs of (a) single annealed ; 1143K and
(b) duplex annealed ; 1143K + 993K in SP-700.